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(54)【発明の名称】 金属精密研磨用レジンボンド砥石

(57)【要約】
【目的】 各種の金属及び合金、特に研磨傷や筋が入り易く、かつうねり面が生じ易い軟質材料に対しても面粗度の優れる鏡面に仕上げることが可能な砥石を提供する。
【構成】 炭酸塩の砥粒を少なくとも３０体積％含有させた金属精密研磨用レジンボンド砥石。
【効果】 従来の砥石に比べて、被加工材料の面粗度が約５～２３０倍も向上し、同一被加工材料の面粗度で比較すると、面粗度で約２０～１６０倍も向上するという効果がある。

【特許請求の範囲】

【請求項１】　炭酸塩の砥粒を少なくとも３０体積％含有させたことを特徴とする金属精密研磨用レジンボンド砥石。

【請求項２】　上記炭酸塩がアルカリ金属及びアルカリ土類金属の中の少なくとも１種の炭酸塩からなることを特徴とする請求項１記載の金属精密研磨用レジンボンド砥石。

【請求項３】　上記炭酸塩が平均粒径５０μm以下の粒子でなることを特徴とする請求項１又は２記載の金属精密研磨用レジンボンド砥石。

【請求項４】　炭酸塩が３０～８０体積％と、樹脂が１５～４９体積％と、残部が炭酸塩を除いた他の無機塩とからなることを特徴とする金属精密研磨用レジンボンド砥石。

【請求項５】　上記炭酸塩がアルカリ金属の炭酸塩，アルカリ土類金属の炭酸塩及び／又はこれらの相互固溶体からなり、上記樹脂がフェノール，エポキシ，ポリイミド，ポリスチレン又はポリエチレンからなることを特徴とする請求項４記載の金属精密研磨用レジンボンド砥石。

【発明の詳細な説明】

【０００１】

【産業上の利用分野】本発明は、半導体用ウェハー，時計枠に代表される装飾品，レーザ用反射鏡，光学部品，精密機械部品，精密計測器部品，電子部品等の各種産業に用いられる金属又は合金の表面を鏡面研磨するのに最適な金属精密研磨用レジンボンド砥石に関するものである。

【０００２】

【従来の技術】従来、研削加工又は研削加工の内、ラッピングやポリシクングのように鏡面研磨を目的とする精密研磨には、被加工材料よりも硬い主としてダイヤモンドや立方晶窒化ホウ素のような超硬質砥粒もしくはそれを含む砥石が用いられている。

【０００３】これに対し、被加工材料よりも軟質な砥粒又はそれを含む砥石を用いて、被加工材料との機械的な作用により誘起される化学反応、所謂メカノケミカルを生じさせる仕上加工法が提案されている。

【０００４】このメカノケミカルによる加工法の代表的なものとしては、Voraらの「Mechanochemical Polishing of SiliconNitride」［J．Am．Cer．Soc，６５（９）C－１４０（１９８２）］、唐木らの「Si単結晶のメカノケミカルポリシングにおける加工速度促進機構」［精密機械，４６，３（１９８０）５３－５９］、落合らの「フェライト材のメカノケミカルポリシング」［精密工学会誌，５４，３（１９８８）１４６－１５１］及び特開昭６４－６４７６６号公報がある。

【０００５】

【発明が解決しようとする課題】Voraらの「Mechanochemical Polishing of Silicon Nitride」には、窒化ケイ素とセラミックスを$CaCO_3$，$MgO$，$SiO_2$，$Fe_2O_3$，$Fe_3O_4$等の軟質な砥粒を用いて湿式でメカノケミカルポリシングをしたことが記載されており、唐木らの「Si単結晶のメカノケミカルポリシングにおける加工速度促進機構」には、Si単結晶をアルカリ性溶液中、$ZrO_2$砥粒によりメカノケミカルポリシングしたことについて記載されている。落合らの「フェライト材のメカノケミカルポリシング」には、Mn－Zn系単結晶フェライトを蒸留水，水酸化ナトリウム，塩酸溶液中、$Al_2O_3$砥粒によりメカノケミカルポリシングしたことについて記載されている。

【０００６】また、特開昭６４－６４７６６号公報には、被加工材料との反応性に優れた金属，酸化物，窒化物，ケイ化物，ホウ化物，炭化物の砥粒と、ポリアセタール，ポリエチレン，アクリロニトリル，ポリフェニルサルファイド，フェノール，エポキシの樹脂とを被加工材料より軟質に成形した砥石材が記載されている。

【０００７】以上の内、Vora，唐木，落合らの前者は、酸化物砥粒を用いてメカノケミカルポリシングを行ったものであり、後者の特開昭６４－６４７６６号公報は、酸化物、特に$Cr_2O_3$，$Fe_2O_3$，$CeO_2$の酸化物砥粒と樹脂とからなる砥石について記載されている。これらに用いられている砥粒は、化学的に安定な金属酸化物からなるために、ポリシング効果が弱く、溶媒を介在させてポリシング効果を高めようとしていること、被加工材料の表面を鏡面にする場合、長時間加工する必要があること、特に、金属又は合金の表面を鏡面状態に仕上加工する場合、砥粒による傷や筋が入り易く、長時間加工しても完全に除去し難く、面にうねりが生じ易くなるという問題がある。

【０００８】本発明は、上述のような問題点を解決したもので、具体的には、炭酸塩の砥粒を樹脂で固めて、ポリシング効果の高い、被加工材料に研磨傷や筋が入り難く、面粗度を高めることが可能な金属精密研磨用レジンボンド砥石の提供を目的とするものである。

【０００９】

【課題を解決するための手段】本発明者らは、半導体用Siウェハー，レーザ用反射鏡又は精密計測器部品等に用いられる金属製の被加工材料の表面を鏡面加工し、加工時間の短縮及び被加工材料の面精度の向上について検討していた所、超硬質砥粒よりも軟質な炭酸塩を樹脂で固めた砥石を用いて、メカノケミカルポリシングを行うと鏡面研磨ができること、このとき市販の金属酸化物を含む砥石が酸性溶液又はアルカリ性溶液を媒介とする湿式によるメカノケミカルポリシングを行う必要があるのに対し、炭酸塩を含む砥石は、乾式でもつてメカノケミカルポリシング効果が十分に生じること、及びその効果

として、被加工材料の表面への影響、例えば、応力，酸素付着，酸化膜付着等が生じなく、かつ砥粒による傷や筋を完全に除去して面精度を顕著に向上させうるという知見を得た。

【００１０】本発明は、上述の知見に基づいて完成したものである。

【００１１】すなわち、本発明の金属精密研磨用レジンボンド砥石は、炭酸塩の砥粒を少なくとも３０体積％含有していることを特徴とするものである。

【００１２】本発明における炭酸塩の砥粒は、具体的には、例えば$Li_2CO_3$，$Na_2CO_3$，$K_2CO_3$，$KHCO_3$，$NaHCO_3$，$Rb_2CO_3$，$Cs_2CO_3$（Na，K）$CO_3$，$MgCO_3$，$CaCO_3$，$SrCO_3$，$BaCO_3$及びこれらの相互固溶体を挙げることができる。これらの内、水や水蒸気との反応が生じ難く、実用上及び品質管理上からも安定性の高いアルカリ土類金属の炭酸塩が特に好ましいものである。

【００１３】炭酸塩の砥粒は、微細である程活性化が高く、被加工材料の面精度の向上が顕著になることから好ましく実質的には平均粒径５０μm以下、好ましくは５μm以下であって、かつ粒度分布の標準偏差がσ以内の均一な粒子からなる砥粒、特に好ましくは平均粒径０．０５μm以下の粒子である。この砥粒が３０体積％未満になると被加工材料の面精度の向上効果が顕著に低下する。

【００１４】本発明の金属精密研磨用レジンボンド砥石は、特に上述の炭酸塩の砥粒が３０～８０体積％と、樹脂が１５～４９体積％と、残部が金属の化合物を主成分とする他物質とからなる場合が砥石の強度上からも好ましいことである。

【００１５】本発明における樹脂は、特に制限を受けるものでないが、フェノール，エポキシ，ポリイミド，ポリスチレン又はポリエチレンでなる場合には、砥石の成形性，強度及び砥石の成形時での砥粒及び他物質の分散性にすぐれるので好ましいことである。

【００１６】本発明における他物質とは、従来から砥石に用いられている物質ならば特に制限を受けるものでなく、具体的には、超硬質砥粒，セラミックス砥粒，ガラス，ウィスカー，繊維，カーボンを挙げることができ、さらに具体的には、例えば$Al_2O_3$，$SiO_2$，$SiC$，$Fe_2O_3$，$Fe_3O_4$，$Cr_2O_3$，$ZrO_2$，$CeO_2$，CBN，WBN，（ウルツ鉱型BN），ダイヤモンドを挙げることができる。また、本発明者が発明し、平成３年６月２７日付で特許出願した砥石に含まれる砥粒を他物質として使用すると、効果が高いものである。

【００１７】本発明の金属精密研磨用レジンボンド砥石は、従来から行われている砥石の成形法、具体的には、砥粒と樹脂又は必要に応じて他物質の含有した出発原料を混合，篩別後、金型でもってホットプレスして作製することができる。

【００１８】

【作用】本発明の金属精密研磨用レジンボンド砥石は、使用時には、炭酸塩の砥粒に内在している活性力と被加工材料の表面に生じる摩擦熱により、砥粒及び被加工材料の表面を原始的溶解状態にさせ、一層活性化を高め、砥粒と被加工材料との化学反応の促進作用、特に炭酸塩の分解による被加工材料の表面エッチング作用、所謂メカノケミカル作用を顕著に生じさせている。また、砥石に含有する樹脂は、炭酸塩の砥粒及び他物質を保持し、砥石の強度を高める作用及び、使用時には、炭酸塩の砥粒と被加工材料の表面とを接触しやすくする作用をしているものである。

【００１９】

【実施例１】各種の炭酸塩の粉末と、各種の樹脂を用いて、表１のように配合し、各試料を得た。

【００２０】表１に示した配合組成の各試料を擂潰機にて混合，篩別後、表２に示す焼結条件でもって砥石を作製した。

【００２１】

【表１】

| 試料番号 | 配合組成 | (VOl%) |
|---|---|---|
| | 砥粒 (平均粒径μm) | 樹脂 (Bal) |
| 本発明品1 | $70SrCO_3$ (10) | フェノール |
| 本発明品2 | $65CaCO_3$ (5) | ポリイミド |
| 本発明品3 | $35BaCO_3$ (2) − $20NaC\ell$ (5) | エポキシ |
| 本発明品4 | $40(K_{0.1},Sr_{0.9})CO_3$ (4) − $15LiF$ (1) | ポリエチレン |
| 本発明品5 | $35SrCO_3$ (3) − $20Cu(NO_3)_2$ (5) | フェノール |
| 本発明品6 | $30(Rb_{0.1},Sr_{0.9})CO_3$ (3) − $Pb(CH_3COO)_2$ (5) | ポリイミド |
| 本発明品7 | $30SrCO_3$ (5) − $20BaCO_3$ − $10KH_3(C_2O_4)$ (10) | ポリエチレ |
| 比較品1 | $95SrCO_3$ (10) | フェノール |
| 比較品2 | $10BaCO_3$ (5) − $5NaC\ell$ (1) | ポリイミド |
| 比較品3 | $50LiF$ (1) − $Pb(CH_3COO)_2$ (5) | エポキシ |

【００２２】

【表２】

| 試料番号 | 焼結条件 | | | |
|---|---|---|---|---|
| | 圧力 $(kg/cm^2)$ | 温度 (℃) | 雰囲気 | 時間 $(h_r)$ |
| 本発明品1 | 250 | 187 | 大気 | 1 |
| 本発明品2 | 250 | 245 | Ar | 3 |
| 本発明品3 | 600 | 195 | $H_2$ | 1 |
| 本発明品4 | 600 | 210 | $N_2$ | 3 |
| 本発明品5 | 250 | 185 | 大気 | 2 |
| 本発明品6 | 250 | 240 | Ar | 2 |
| 本発明品7 | 550 | 205 | 大気 | 2 |
| 比較品1 | 200 | 180 | 大気 | 1 |
| 比較品2 | 220 | 230 | Ar | 2 |
| 比較品3 | 600 | 140 | $H_2$ | 1 |

表１及び表２により得られた本発明品１～７と比較品１～３に市販のシリカゾルを比較品４として、下記の条件でもって鏡面研磨仕上を試みた。

鏡面研磨仕上げ条件

被加工材料：表３に示す各種金属又は合金の表面を１μmダイヤモンド砥石で研磨した面、

回転数　：２００ｒｐｍ

加工圧力　：１５０ｇ／ｃｍ$^2$

研磨方式　：乾式

評価　：所定の研磨時間における被加工材料の面粗度、６０分研磨後における被加工材料の面状態及び砥石の損傷状態の観察。

【００２３】上記鏡面研磨仕上試験の結果を表３に示した。

【００２４】

【表３】

| 試料番号 | | 被加工材料 | 各研磨時間（分）毎における面粗度 Rmax（nm） | | | | 60分研磨後の状態 | | 被摩耗量 |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | 被加工材料の表面 | 砥石の損傷状態 | m㎡/ kgf・mm |
| | | | 10 | 30 | 50 | 60 | | | |
| 本発明品 | 1 | Siウエハー | 12 | 7 | 3 | 2 | 鏡面 | 平面度良好 | 2×10⁻⁵ |
| | 2 | Al合金 | 2 | 1.3 | 0.7 | 0.5 | 鏡面 | 平面度良好 | 9×10⁻⁷ |
| | 3 | 工具鋼 | 2.4 | 1.5 | 0.9 | 0.8 | 鏡面 | 平面度良好 | 6×10⁻⁶ |
| | 4 | 浸炭鋼 | 1.5 | 0.9 | 0.7 | 0.6 | 鏡面 | 平面度良好 | 2×10⁻⁷ |
| | 5 | 銅合金 | 2.3 | 1.7 | 1.3 | 1.0 | 鏡面 | 平面度良好 | 1×10⁻⁶ |
| | 6 | Ti合金 | 2.3 | 1.9 | 1.5 | 1.1 | 鏡面 | 平面度良好 | 3×10⁻⁵ |
| | 7 | Au合金 | 2.5 | 1.9 | 1.0 | 0.9 | 鏡面 | 平面度良好 | 6×10⁻⁷ |
| 比較品 | 1 | Siウエハー | 150 | 129 | 107 | 103 | 傷,くもり有り | 粒子脱落 | 3×10⁻¹⁰ |
| | 2 | Al合金 | 148 | 137 | 128 | 121 | 傷,くもり有り | 粒子脱落 | 2×10⁻⁹ |
| | 3 | 工具鋼 | 36 | 29 | 23 | 23 | 傷,くもり有り | 粒子脱落 | 1×10⁻⁶ |
| | 4 | Siウエハー | 42 | 20 | 13 | 10 | 傷,くもり有り | 粒子脱落 | 7×10⁻⁹ |

【0025】

【発明の効果】本発明の金属精密研磨用レジンボンド砥石は、砥石中に含有している炭酸塩の砥粒と、使用時に被加工材料の表面に発生する摩擦熱でもって、被加工材料の表面でサブナノメーターのオーダーでの溶解及び酸化，酸化被膜形成及びその被膜除去が繰り返されるという、酸化反応の促進効果の高い砥石であり、従来の砥粒を含有した砥石に比べて、被加工材料の面粗度（Ｒｍａｘ）において、約５～２３０倍も向上させうるという顕著な効果を有するものである。

JAPANESE [JP,05-050379,A]

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CLAIMS DETAILED DESCRIPTION TECHNICAL FIELD PRIOR ART EFFECT OF THE INVENTION TECHNICAL PROBLEM MEANS OPERATION

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## CLAIMS

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[Claim(s)]
[Claim 1] The resin bond grinding stone for metal precision polish characterized by doing 30 volume % content of the abrasive grain of a carbonate at least.
[Claim 2] The resin bond grinding stone for metal precision polish according to claim 1 characterized by the above-mentioned carbonate consisting of at least one sort of carbonates in alkali metal and alkaline earth metal.
[Claim 3] The resin bond grinding stone for metal precision polish according to claim 1 or 2 characterized by the above-mentioned carbonate becoming by the particle with a mean particle diameter of 50 micrometers or less.
[Claim 4] The resin bond grinding stone for metal precision polish with which a carbonate is characterized by consisting of 30 - 80 volume % and other mineral salt excluding [ 15 - 49 volume % and the remainder ] the carbonate in resin.
[Claim 5] The resin bond grinding stone for metal precision polish according to claim 4 characterized by for the above-mentioned carbonate consisting of the carbonate of alkali metal, carbonates of alkaline earth metal, and/or these mutual solid solutions, and the above-mentioned resin consisting of a phenol, epoxy, polyimide, polystyrene, or polyethylene.

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## DETAILED DESCRIPTION

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[Detailed Description of the Invention]
[0001]
[Industrial Application] This invention relates to the optimal resin bond grinding stone for metal precision polish for carrying out mirror polishing of the front face of the metal used for various industries, such as the wafer for semi-conductors, the accessories represented by the clock frame, a reflecting mirror for laser, an optic, a precision machinery component, precision measuring instrument components, and electronic parts, or an alloy.
[0002]
[Description of the Prior Art] Conventionally, the grinding stone containing a nature abrasive grain harder than a work material of superhard mainly like a diamond or cubic boron nitride or it is used for precision polish aiming at mirror polishing like wrapping or polish KUNGU among the grinding process or the grinding process.
[0003] On the other hand, the chemical reaction in which induction is carried out by mechanical operation with a work material, and the finish method for producing the so-called MEKANOKEMIKARU are proposed using the grinding stone containing an abrasive grain or it elasticity [ work material ].
[0004] As a typical thing of this processing method depended mechanochemical Vora's and others "Mechanochemical Polishing of SiliconNitride" [J. Am.Cer.Soc, 65(9) C-140(1982)], Karaki's and others "working speed acceleration device in the mechanochemical polishing of Si single crystal" [a precision instrument, 46, 3 (1980)53-59], There are "the mechanochemical polishing of ferrite material" [the Japan Society for precision Engineering, 54, 3 (1988)146-151] and JP,64-64766,A of Ochiai and others.
[0005]
[Problem(s) to be Solved by the Invention] Having carried out mechanochemical polishing with wet using the elasticity grade [ CaCO3, MgO, SiO2, Fe 2O3, and Fe3O4 grade ] abrasive grain is indicated in silicon nitride and the ceramics by Vora's and others "Mechanochemical Polishing ofSilicon Nitride", and having carried out mechanochemical polishing of the Si single crystal to the Karaki's and others "working speed acceleration device in the mechanochemical polishing of Si single crystal" with ZrO2 abrasive grain among the alkaline solution is indicated. Having carried out mechanochemical polishing of the Mn-Zn system single crystal ferrite with aluminum2O3 abrasive grain among distilled water, a sodium hydroxide, and the solution of hydrochloric acid is indicated by "the mechanochemical polishing of ferrite material" of Ochiai and others.
[0006] Moreover, the grinding stone material which fabricated the abrasive grain of the metal excellent in reactivity with a work material, oxide, a nitride, a silicide, a boride, and carbide and the resin of polyacetal, polyethylene, acrylonitrile, polyphenyl sulfide, a phenol, and epoxy from the work material to elasticity is indicated by JP,64-64766,A.
[0007] The former of Vora, Karaki, Ochiai and others performs mechanochemical polishing among more than using an oxide abrasive grain, and latter JP,64-64766,A is indicated about the grinding stone which consists of an oxide especially Cr 2O3, Fe2O3, and the oxide abrasive grain and resin of CeO2. Since the abrasive grain used for these consists of a stable metallic oxide

chemically The polishing effectiveness tends to be weak, tends to make a solvent intervene, and tends to heighten the polishing effectiveness, That it is necessary to carry out long duration processing and when making the front face of a work material into a mirror plane, and changing the finish of the front face of a metal or an alloy into a mirror plane condition especially, even if the blemish and muscle by the abrasive grain long-duration-processing-make it easy to enter, it is hard to remove thoroughly, and the problem of becoming easy to produce a wave is in a field.
[0008] It is what solved the above troubles, and this invention hardens the abrasive grain of a carbonate by resin, and neither a polish blemish nor a muscle can go into a work material with the high polishing effectiveness easily, and it aims concrete at offer of the resin bond grinding stone for metal precision polish which can raise field relative roughness.
[0009]
[Means for Solving the Problem] this invention persons do mirror plane processing of the front face of the metal work material used for Si wafer for semi-conductors, the reflecting mirror for laser, or precision measuring instrument components. Using the place which was considering compaction of floor to floor time, and improvement in the profile irregularity of a work material, and the grinding stone which hardened the carbonate [ elasticity / abrasive grain / of superhard / nature ] by resin, if mechanochemical polishing is performed, mirror polishing will be made, The grinding stone with which the grinding stone which contains a commercial metallic oxide at this time contains a carbonate to performing mechanochemical polishing by wet [ through an acidic solution or an alkaline solution ] The knowledge of the effect of the front face on a work material, for example, stress, oxygen adhesion, oxide-film adhesion, etc. not arising, and having removed the blemish and muscle by the abrasive grain thoroughly as the intermediary mechanochemical polishing effectiveness also fully producing dry type and its effectiveness, having raised profile irregularity notably, and getting was acquired.
[0010] This invention is completed based on above-mentioned knowledge.
[0011] That is, the resin bond grinding stone for metal precision polish of this invention is characterized for the abrasive grain of a carbonate by carrying out 30 volume % content at least.

[0012] Specifically, the abrasive grain of the carbonate in this invention can mention Li2CO3, Na2CO3, K2CO3, KHCO3 and NaHCO3, Rb2CO3, Cs2CO3(Na, K) CO3, MgCO3, CaCO3, SrCO3 and BaCO3, and these mutual solid solutions. Among these, it is hard to produce a reaction with water or a steam, and reaches practically, and the carbonate of an extremely stable alkaline earth metal is especially desirable also from quality control.
[0013] the abrasive grain with which it is 5 micrometers or less preferably the mean particle diameter of 50 micrometers or less preferably and substantially from the abrasive grain of a carbonate having activation so high that it being detailed, and improvement in the profile irregularity of a work material becoming remarkable, and the standard deviation of particle size distribution consists of a uniform particle within sigma -- it is a particle with a mean particle diameter of 0.05 micrometers or less especially preferably. If this abrasive grain becomes under 30 volume %, the improvement effectiveness of the profile irregularity of a work material will fall notably.
[0014] The resin bond grinding stone for metal precision polish of this invention is that the case where 15 - 49 volume % and the remainder use [ 30 - 80 volume % and resin ] a metaled compound as a principal component, and also the abrasive grain of an above-mentioned carbonate consists of matter especially is desirable also from the reinforcement of a grinding stone.
[0015] Although a limit is not received, especially the resin in this invention is a desirable thing since it excels in the dispersibility of the abrasive grain in the time of shaping of the moldability of a grinding stone, reinforcement, and a grinding stone, and other matter, when becoming with a phenol, epoxy, polyimide, polystyrene, or polyethylene.
[0016] It is not what will receive a limit especially if it is the matter with which the matter is used for the grinding stone from the former also in this invention. Specifically The nature abrasive grain of superhard, a ceramic abrasive grain, glass, a whisker, fiber, and carbon can be mentioned. Still more specifically For example, aluminum 2O3, SiO2, SiC and Fe 2O3, Fe3O4,

Cr2O3, ZrO2, CeO2, CBN and WBN, the (wurtzite mold BN), and a diamond can be mentioned. Moreover, effectiveness is high when the abrasive grain contained in the grinding stone which this invention person invented and carried out patent application on June 27, Heisei 3 is used as other matter.

[0017] The hotpress of the resin bond grinding stone for metal precision polish of this invention can be carried out to the method of fabricating the grinding stone currently performed from the former, and it being also with after [ according to mixing and screen ] and metal mold about the start raw material which other matter specifically contained an abrasive grain, resin, or if needed, and it can be produced.

[0018]

[Function] With the frictional heat produced on the front face of the activity force inherent in the abrasive grain of a carbonate, and a work material, at the time of an activity, the resin bond grinding stone for metal precision polish of this invention makes the front face of an abrasive grain and a work material change into a primitive dissolution condition, raises activation further, and is producing notably the acceleration operation of the chemical reaction of an abrasive grain and a work material especially the surface etching operation of the work material by disassembly of a carbonate, and the so-called mechanochemical operation at it. Moreover, the resin contained in a grinding stone holds the abrasive grain and other matter of a carbonate, and is carrying out the operation make it easy to contact the abrasive grain of a carbonate, and the front face of a work material at the time of the operation which raises the reinforcement of a grinding stone, and an activity.

[0019]

[Example 1] Using the powder of various kinds of carbonates, and various kinds of resin, it blended, as shown in a table 1, and each sample was obtained.

[0020] The grinding stone was produced as it is also at the sintering conditions which show each sample of the combination presentation shown in a table 1 in a table 2 the back according to mixing and screen with a **** machine.

[0021]

[A table 1]

| 試料番号 | 配　合　組　成　(VOl%) | |
|---|---|---|
| | 砥　粒　(平均粒径μm) | 樹脂 (Bal) |
| 本発明品1 | $70SrCO_3$ (10) | フェノール |
| 本発明品2 | $65CaCO_3$ (5) | ポリイミド |
| 本発明品3 | $35BaCO_3$ (2) −20NaCℓ (5) | エポキシ |
| 本発明品4 | 40 $(K_{0.1},Sr_{0.9})CO_3$ (4) −15LiF (1) | ポリエチレン |
| 本発明品5 | $35SrCO_3$ (3) −20Cu $(NO_3)_2$ (5) | フェノール |
| 本発明品6 | 30 $(Rb_{0.1},Sr_{0.9})CO_3$ (3) −Pb $(CH_3COO)_2$ (5) | ポリイミド |
| 本発明品7 | $30SrCO_3$ (5) −$20BaCO_3$ − $10KH_3(C_2O_4)$ (10) | ポリエチレ |
| 比較品1 | $95SrCO_3$ (10) | フェノール |
| 比較品2 | $10BaCO_3$ (5) −5NaCℓ (1) | ポリイミド |
| 比較品3 | 50LiF (1) −Pb $(CH_3COO)_2$ (5) | エポキシ |

[0022]

[A table 2]

| 試料番号 | 焼結条件 | | | |
|---|---|---|---|---|
| | 圧力 (kg/cm$^2$) | 温度 (℃) | 雰囲気 | 時間 (hr) |
| 本発明品1 | 250 | 187 | 大気 | 1 |
| 本発明品2 | 250 | 245 | Ar | 3 |
| 本発明品3 | 600 | 195 | $H_2$ | 1 |
| 本発明品4 | 600 | 210 | $N_2$ | 3 |
| 本発明品5 | 250 | 185 | 大気 | 2 |
| 本発明品6 | 250 | 240 | Ar | 2 |
| 本発明品7 | 550 | 205 | 大気 | 2 |
| 比較品1 | 200 | 180 | 大気 | 1 |
| 比較品2 | 220 | 230 | Ar | 2 |
| 比較品3 | 600 | 140 | $H_2$ | 1 |

By using as the comparison article 4 the silica sol of marketing in this invention article 1-7 and the comparison article 1-3 which were obtained by the table 1 and the table 2, mirror-polishing finish was tried as it is also at the following conditions.
Mirror-polishing finishing condition work material: The field, rotational frequency which ground the front face of the various metals shown in a table 3, or an alloy by 1-micrometer diamond wheel : 200rpm processing pressure force : 150 g/cm2 polish method : Dry type assessment : Observation of the field condition of the field relative roughness of the work material in predetermined polish time amount, and the work material after 60-minute polish, and the faulted condition of a grinding stone.
[0023] The result of the above-mentioned mirror-polishing finish trial was shown in a table 3.
[0024]
[A table 3]

| 試料番号 | | 被加工材料 | 各研磨時間（分）毎における面粗度 Rmax（nm） | | | | ６０分研磨後の状態 | | 被摩耗量 |
|---|---|---|---|---|---|---|---|---|---|
| | | | 10 | 30 | 50 | 60 | 被加工材料の表面 | 砥石の損傷状態 | m㎥／kgf・mm |
| 本発明品 | 1 | Siウエハー | 12 | 7 | 3 | 2 | 鏡面 | 平面度良好 | $2 \times 10^{-5}$ |
| | 2 | Al合金 | 2 | 1.3 | 0.7 | 0.5 | 鏡面 | 平面度良好 | $9 \times 10^{-7}$ |
| | 3 | 工具鋼 | 2.4 | 1.5 | 0.9 | 0.8 | 鏡面 | 平面度良好 | $6 \times 10^{-6}$ |
| | 4 | 浸炭鋼 | 1.5 | 0.9 | 0.7 | 0.6 | 鏡面 | 平面度良好 | $2 \times 10^{-7}$ |
| | 5 | 銅合金 | 2.3 | 1.7 | 1.3 | 1.0 | 鏡面 | 平面度良好 | $1 \times 10^{-6}$ |
| | 6 | Ti合金 | 2.3 | 1.9 | 1.5 | 1.1 | 鏡面 | 平面度良好 | $3 \times 10^{-5}$ |
| | 7 | Au合金 | 2.5 | 1.9 | 1.0 | 0.9 | 鏡面 | 平面度良好 | $6 \times 10^{-7}$ |
| 比較品 | 1 | Siウエハー | 150 | 129 | 107 | 103 | 傷,くもり有り | 粒子脱落 | $3 \times 10^{-10}$ |
| | 2 | Al合金 | 148 | 137 | 128 | 121 | 傷,くもり有り | 粒子脱落 | $2 \times 10^{-9}$ |
| | 3 | 工具鋼 | 36 | 29 | 23 | 23 | 傷,くもり有り | 粒子脱落 | $1 \times 10^{-8}$ |
| | 4 | Siウエハー | 42 | 20 | 13 | 10 | 傷,くもり有り | 粒子脱落 | $7 \times 10^{-9}$ |

[0025]
[Effect of the Invention] The resin bond grinding stone for metal precision polish of this invention It has with the abrasive grain of the carbonate contained in a grinding stone, and the frictional heat generated on the surface of a work material at the time of an activity. Say that the dissolution and oxidation, oxide skin formation, and its coat clearance of SABUNANOMETA to order are repeated on the surface of a work material. It is the high grinding stone of the facilitatory effect of oxidation reaction, and has the remarkable effectiveness of making it improving also about five to 230 times, and getting in the field relative roughness (Rmax) of a work material compared with the grinding stone containing the conventional abrasive grain.

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## TECHNICAL FIELD

[Industrial Application] This invention relates to the optimal resin bond grinding stone for metal precision polish for carrying out mirror polishing of the front face of the metal used for various industries, such as the wafer for semi-conductors, the accessories represented by the clock frame, a reflecting mirror for laser, an optic, a precision machinery component, precision measuring instrument components, and electronic parts, or an alloy.

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## PRIOR ART

[Description of the Prior Art] Conventionally, the grinding stone containing a nature abrasive grain harder than a work material of superhard mainly like a diamond or cubic boron nitride or it is used for precision polish aiming at mirror polishing like wrapping or polish KUNGU among the grinding process or the grinding process.
[0003] On the other hand, the chemical reaction in which induction is carried out by mechanical operation with a work material, and the finish method for producing the so-called MEKANOKEMIKARU are proposed using the grinding stone containing an abrasive grain or it elasticity [ work material ].
[0004] As a typical thing of this processing method depended mechanochemical Vora's and others "Mechanochemical Polishing of SiliconNitride" [J. Am.Cer.Soc, 65(9) C-140(1982)], Karaki's and others "working speed acceleration device in the mechanochemical polishing of Si single crystal" [a precision instrument, 46, 3 (1980)53-59], There are "the mechanochemical polishing of ferrite material" [the Japan Society for precision Engineering, 54, 3 (1988)146-151] and JP,64-64766,A of Ochiai and others.
[0005]

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## EFFECT OF THE INVENTION

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[Effect of the Invention] The resin bond grinding stone for metal precision polish of this invention It has with the abrasive grain of the carbonate contained in a grinding stone, and the frictional heat generated on the surface of a work material at the time of an activity. Say that the dissolution and oxidation, oxide skin formation, and its coat clearance of SABUNANOMETA to order are repeated on the surface of a work material. It is the high grinding stone of the facilitatory effect of oxidation reaction, and has the remarkable effectiveness of making it improving also about five to 230 times, and getting in the field relative roughness (Rmax) of a work material compared with the grinding stone containing the conventional abrasive grain.

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## TECHNICAL PROBLEM

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[Problem(s) to be Solved by the Invention] Having carried out mechanochemical polishing with wet using the elasticity grade [ $CaCO_3$, MgO, $SiO_2$, $Fe_2O_3$, and $Fe_3O_4$ grade ] abrasive grain is indicated in silicon nitride and the ceramics by Vora's and others "Mechanochemical Polishing ofSilicon Nitride", and having carried out mechanochemical polishing of the Si single crystal to the Karaki's and others "working speed acceleration device in the mechanochemical polishing of Si single crystal" with $ZrO_2$ abrasive grain among the alkaline solution is indicated. Having carried out mechanochemical polishing of the Mn-Zn system single crystal ferrite with aluminum2O3 abrasive grain among distilled water, a sodium hydroxide, and the solution of hydrochloric acid is indicated by "the mechanochemical polishing of ferrite material" of Ochiai and others.

[0006] Moreover, the grinding stone material which fabricated the abrasive grain of the metal excellent in reactivity with a work material, oxide, a nitride, a silicide, a boride, and carbide and the resin of polyacetal, polyethylene, acrylonitrile, polyphenyl sulfide, a phenol, and epoxy from the work material to elasticity is indicated by JP,64-64766,A.

[0007] The former of Vora, Karaki, Ochiai and others performs mechanochemical polishing among more than using an oxide abrasive grain, and latter JP,64-64766,A is indicated about the grinding stone which consists of an oxide especially $Cr_2O_3$, $Fe_2O_3$, and the oxide abrasive grain and resin of $CeO_2$. Since the abrasive grain used for these consists of a stable metallic oxide chemically The polishing effectiveness tends to be weak, tends to make a solvent intervene, and tends to heighten the polishing effectiveness, That it is necessary to carry out long duration processing and when making the front face of a work material into a mirror plane, and changing the finish of the front face of a metal or an alloy into a mirror plane condition especially, even if the blemish and muscle by the abrasive grain long-duration-processing-make it easy to enter, it is hard to remove thoroughly, and the problem of becoming easy to produce a wave is in a field.

[0008] It is what solved the above troubles, and this invention hardens the abrasive grain of a carbonate by resin, and neither a polish blemish nor a muscle can go into a work material with the high polishing effectiveness easily, and it aims concrete at offer of the resin bond grinding stone for metal precision polish which can raise field relative roughness.

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## MEANS

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[Means for Solving the Problem] this invention persons do mirror plane processing of the front face of the metal work material used for Si wafer for semi-conductors, the reflecting mirror for laser, or precision measuring instrument components. Using the place which was considering compaction of floor to floor time, and improvement in the profile irregularity of a work material, and the grinding stone which hardened the carbonate [ elasticity / abrasive grain / of superhard / nature ] by resin, if mechanochemical polishing is performed, mirror polishing will be made, The grinding stone with which the grinding stone which contains a commercial metallic oxide at this time contains a carbonate to performing mechanochemical polishing by wet [ through an acidic solution or an alkaline solution ] The knowledge of the effect of the front face on a work material, for example, stress, oxygen adhesion, oxide-film adhesion, etc. not arising, and having removed the blemish and muscle by the abrasive grain thoroughly as the intermediary mechanochemical polishing effectiveness also fully producing dry type and its effectiveness, having raised profile irregularity notably, and getting was acquired.
[0010] This invention is completed based on above-mentioned knowledge.
[0011] That is, the resin bond grinding stone for metal precision polish of this invention is characterized for the abrasive grain of a carbonate by carrying out 30 volume % content at least.

[0012] Specifically, the abrasive grain of the carbonate in this invention can mention $Li_2CO_3$, $Na_2CO_3$, $K_2CO_3$, $KHCO_3$ and $NaHCO_3$, $Rb_2CO_3$, $Cs_2CO_3$(Na, K) $CO_3$, $MgCO_3$, $CaCO_3$, $SrCO_3$ and $BaCO_3$, and these mutual solid solutions. Among these, it is hard to produce a reaction with water or a steam, and reaches practically, and the carbonate of an extremely stable alkaline earth metal is especially desirable also from quality control.
[0013] the abrasive grain with which it is 5 micrometers or less preferably the mean particle diameter of 50 micrometers or less preferably and substantially from the abrasive grain of a carbonate having activation so high that it being detailed, and improvement in the profile irregularity of a work material becoming remarkable, and the standard deviation of particle size distribution consists of a uniform particle within sigma — it is a particle with a mean particle diameter of 0.05 micrometers or less especially preferably. If this abrasive grain becomes under 30 volume %, the improvement effectiveness of the profile irregularity of a work material will fall notably.
[0014] The resin bond grinding stone for metal precision polish of this invention is that the case where 15 - 49 volume % and the remainder use [ 30 - 80 volume % and resin ] a metaled compound as a principal component, and also the abrasive grain of an above-mentioned carbonate consists of matter especially is desirable also from the reinforcement of a grinding stone.
[0015] Although a limit is not received, especially the resin in this invention is a desirable thing since it excels in the dispersibility of the abrasive grain in the time of shaping of the moldability of a grinding stone, reinforcement, and a grinding stone, and other matter, when becoming with a phenol, epoxy, polyimide, polystyrene, or polyethylene.
[0016] It is not what will receive a limit especially if it is the matter with which the matter is used for the grinding stone from the former also in this invention. Specifically The nature

abrasive grain of superhard, a ceramic abrasive grain, glass, a whisker, fiber, and carbon can be mentioned. Still more specifically For example, aluminum 2O3, SiO2, SiC and Fe 2O3, Fe3O4, Cr2O3, ZrO2, CeO2, CBN and WBN, the (wurtzite mold BN), and a diamond can be mentioned. Moreover, effectiveness is high when the abrasive grain contained in the grinding stone which this invention person invented and carried out patent application on June 27, Heisei 3 is used as other matter.

[0017] The hotpress of the resin bond grinding stone for metal precision polish of this invention can be carried out to the method of fabricating the grinding stone currently performed from the former, and it being also with after [ according to mixing and screen ] and metal mold about the start raw material which other matter specifically contained an abrasive grain, resin, or if needed, and it can be produced.

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## OPERATION

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[Function] With the frictional heat produced on the front face of the activity force inherent in the abrasive grain of a carbonate, and a work material, at the time of use, the resin bond grinding stone for metal precision polish of this invention makes the front face of an abrasive grain and a work material change into a primitive dissolution condition, raises activation further, and is producing notably the promotion operation of the chemical reaction of an abrasive grain and a work material especially the surface etching operation of the work material by decomposition of a carbonate, and the so-called mechanochemical operation at it. Moreover, the resin contained in a grinding stone holds the abrasive grain and other matter of a carbonate, and is carrying out the operation make it easy to contact the abrasive grain of a carbonate, and the front face of a work material at the time of the operation which raises the reinforcement of a grinding stone, and use.
[0019]
[Example 1] Using the powder of various kinds of carbonates, and various kinds of resin, it blended, as shown in Table 1, and each sample was obtained.
[0020] The grinding stone was produced as it is also at the sintering conditions which show each sample of the combination presentation shown in Table 1 in Table 2 the back according to mixing and screen with a **** machine.
[0021]
[Table 1]

| 試料番号 | 配合組成 | (VOl%) |
|---|---|---|
| | 砥粒 (平均粒径μm) | 樹脂 (Bal) |
| 本発明品1 | $70SrCO_3$ (10) | フェノール |
| 本発明品2 | $65CaCO_3$ (5) | ポリイミド |
| 本発明品3 | $35BaCO_3$ (2) − $20NaC\ell$ (5) | エポキシ |
| 本発明品4 | 40 $(K_{0.1},Sr_{0.9})$ $CO_3$ (4) − 15LiF (1) | ポリエチレン |
| 本発明品5 | $35SrCO_3$ (3) − 20Cu $(NO_3)_2$ (5) | フェノール |
| 本発明品6 | 30 $(Rb_{0.1},Sr_{0.9})$ $CO_3$ (3) − Pb $(CH_3COO)_2$ (5) | ポリイミド |
| 本発明品7 | $30SrCO_3$ (5) − $20BaCO_3$ − $10KH_3$ $(C_2O_4)$ (10) | ポリエチレ |
| 比較品1 | $95SrCO_3$ (10) | フェノール |
| 比較品2 | $10BaCO_3$ (5) − $5NaC\ell$ (1) | ポリイミド |
| 比較品3 | 50LiF (1) − Pb $(CH_3COO)_2$ (5) | エポキシ |

[0022]

[Table 2]

| 試料番号 | 焼結条件 | | | |
|---|---|---|---|---|
| | 圧力 ($kg/cm^2$) | 温度 (℃) | 雰囲気 | 時間 ($h_r$) |
| 本発明品 1 | 250 | 187 | 大気 | 1 |
| 本発明品 2 | 250 | 245 | Ar | 3 |
| 本発明品 3 | 600 | 195 | $H_2$ | 1 |
| 本発明品 4 | 600 | 210 | $N_2$ | 3 |
| 本発明品 5 | 250 | 185 | 大気 | 2 |
| 本発明品 6 | 250 | 240 | Ar | 2 |
| 本発明品 7 | 550 | 205 | 大気 | 2 |
| 比較品 1 | 200 | 180 | 大気 | 1 |
| 比較品 2 | 220 | 230 | Ar | 2 |
| 比較品 3 | 600 | 140 | $H_2$ | 1 |

By using as the comparison article 4 the silica sol of marketing in this invention article 1-7 and the comparison article 1-3 which were obtained by Table 1 and 2, mirror-polishing finish was tried as it is also at the following conditions.
Mirror-polishing finishing condition work material: The field, rotational frequency which ground the front face of the various metals shown in Table 3, or an alloy by 1-micrometer diamond wheel : 200rpm processing pressure force : 150 g/cm2 polish method : Dry type evaluation : Observation of the field condition of the field roughness of the work material in predetermined polish time amount, and the work material after 60-minute polish, and the faulted condition of a grinding stone.
[0023] The result of the above-mentioned mirror-polishing finish trial was shown in Table 3.
[0024]
[Table 3]

| 試料番号 | | 被加工材料 | 各研磨時間（分）毎における面粗度 Rmax（nm） | | | | ６０分研磨後の状態 | | 被摩耗量 |
|---|---|---|---|---|---|---|---|---|---|
| | | | 10 | 30 | 50 | 60 | 被加工材料の表面 | 砥石の損傷状態 | m㎡／kgf・mm |
| 本発明品 | 1 | Siウエハー | 12 | 7 | 3 | 2 | 鏡面 | 平面度良好 | 2×10⁻⁵ |
| | 2 | Al合金 | 2 | 1.3 | 0.7 | 0.5 | 鏡面 | 平面度良好 | 9×10⁻⁷ |
| | 3 | 工具鋼 | 2.4 | 1.5 | 0.9 | 0.8 | 鏡面 | 平面度良好 | 6×10⁻⁶ |
| | 4 | 浸炭鋼 | 1.5 | 0.9 | 0.7 | 0.6 | 鏡面 | 平面度良好 | 2×10⁻⁷ |
| | 5 | 銅合金 | 2.3 | 1.7 | 1.3 | 1.0 | 鏡面 | 平面度良好 | 1×10⁻⁶ |
| | 6 | Ti合金 | 2.3 | 1.9 | 1.5 | 1.1 | 鏡面 | 平面度良好 | 3×10⁻⁵ |
| | 7 | Au合金 | 2.5 | 1.9 | 1.0 | 0.9 | 鏡面 | 平面度良好 | 6×10⁻⁷ |
| 比較品 | 1 | Siウエハー | 150 | 129 | 107 | 103 | 傷,くもり有り | 粒子脱落 | 3×10⁻¹⁰ |
| | 2 | Al合金 | 148 | 137 | 128 | 121 | 傷,くもり有り | 粒子脱落 | 2×10⁻⁹ |
| | 3 | 工具鋼 | 36 | 29 | 23 | 23 | 傷,くもり有り | 粒子脱落 | 1×10⁻⁸ |
| | 4 | Siウエハー | 42 | 20 | 13 | 10 | 傷,くもり有り | 粒子脱落 | 7×10⁻⁹ |

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[Translation done.]